



Mr. Hauptmann

EXHIBIT 5

100+



Europäisches Patentamt

European Patent Office

Office européen des brevets



⑪ Publication number: 0 389 461 B1

⑫

EUROPEAN PATENT SPECIFICATION

⑯ Date of publication of patent specification :
03.11.93 Bulletin 93/44

⑮ Int. Cl.⁵: A61C 5/10

⑯ Application number : 90850107.5

⑯ Date of filing : 20.03.90

⑯ Artificial onlay tooth crowns and inlays.

⑯ Priority : 23.03.89 SE 8901049

⑯ Date of publication of application :
26.09.90 Bulletin 90/39

⑯ Publication of the grant of the patent :
03.11.93 Bulletin 93/44

⑯ Designated Contracting States :
AT CH DE DK ES FR GB IT LI SE

⑯ References cited :
EP-A- 0 033 492
DE-A- 3 804 531
DE-A- 3 720 308
FR-A- 2 318 842
FR-A- 2 490 950
FR-A- 2 558 717
WO 87/06451, EP-A-0 225 279, EP-A-0 384 907
(Art. 54(3)(4) EPC), Hawley's Condensed
Chemical Dictionary

⑯ Proprietor : SANDVIK AKTIEBOLAG
S-811 81 Sandviken 1 (SE)
Proprietor : Nobelpharma AB
Box 5190
S-402 26 Göteborg (SE)

⑯ Inventor : Odén, Agneta
Bergstigen 26
S-182 74 Stockaund (SE)
Inventor : Andersson, Matts
Hammarströms Täppa 2
S-443 39 Lerum (SE)

⑯ Representative : Östlund, Alf Olof Anders et al
Sandvik AB Patent Department
S-811 81 Sandviken (SE)

EP 0 389 461 B1

Note : Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid (Art. 99(1) European patent convention).

The ceramic powder can be made by several well known methods. Traditional powder metallurgical techniques can be used, where the different components are mixed and ground under dry or wet conditions with water or an inorganic solvent (e.g. alcohols) as grinding liquid. So called SOL-GEL technique can also be used where different oxide materials are deposited together from a water solution or are co precipitated from metal alcoxides in e.g. water free alcohol by controlled addition of water. A combination of different techniques can also be used by using SOL-GEL technique to deposit a surface layer of desired metal oxide on a powder material. Lubricants or other organic binders depending on the choice of forming method may be added to the ceramic powder when needed at suitable times in the process as is conventionally known. Other preparation routes of the ceramic material are also possible such as reaction sintering where a suitable metal is oxidized, nitrided etc. For example, aluminium can be oxidized under carefully controlled processing to alumina. These methods allow preforming or reinforcement by fibres, e.g., in a felt infiltrated with liquid metal.

Many of the monolithic ceramics which are biocompatible may have a brittle performance if they are not sintered to nearly full density, more than 98% and preferably > 99.5% of the theoretical density. However, these ceramics can be strengthened by a number of toughening mechanisms. Finely dispersed particles, platelets, whiskers or fibers raise the fracture toughness of the composite. Typical additives are the nitrides, carbides, borides or mixtures thereof of the transition metal of group IV-VI or of the elements Al or Si. Toughening may also be achieved by so called transformation toughening, i.e., additions of unstabilized ZrO_2 or ZrO_2 stabilized with Y_2O_3 , MgO or CaO . The additions of these latter oxides shall not exceed 25 wt%, but should be more than 2 wt%.

The powder with lubricants and/or other organic binders is cold isostatically compacted, uniaxially pressed, slip cast, pressure cast, injection moulded or compacted in another suitable way. The compacted body has such dimensions that it comprises enough material for the copy milling of the outer shape of the core, which will fit into the prepared cavity (E). During this copy milling the sintering shrinkage must be considered. Thus, the copy milled surface (D) must be enlarged so that the compacted body has such dimensions that after the shrinkage during the subsequent sintering process to high density, has desired final geometrical external shape, (D) which will fit into the prepared cavity (E) with great accuracy.

The ceramic body can also be presintered before the copy milling of the surface (D) fitting to the prepared cavity (E). All the other surfaces are prepared near final shape before the final sintering. It is important that the ceramic material is sintered to closed porosity, which for an oxide material means at least 95% of theoretical density, but in order to ensure good mechanical strength the material should preferably have a density over 98%, while densities over 99.5% give the best strength.

The sintering can take place in a vacuum or under hydrogen atmosphere, under normal atmospheric pressure or under increased pressure in connection with the overpressure sintering or hot isostatic compaction or alternatively by hot pressing. Highly pure Al_2O_3 becomes translucent during sintering to full density in vacuum or in hydrogen atmosphere, which is an advantage when natural teeth are to be imitated. Pure oxide material can be sintered in air, but some composites have to be sintered in inert or controlled atmosphere. The core is given an external shape so that the building-up of the veneer is facilitated. The external shape can be such that it is roughly similar to natural teeth. After the final sintering the surfaces of the core may need some grinding, especially the external surfaces outside the prepared cavity. This grinding will be made with the inlay or the onlay crown on a model of the prepared tooth. If dental porcelain is used as the veneer with a coefficient of thermal expansion adapted to the material of the core, the porcelain will adhere better. In the case of Al_2O_3 as the core material there will be a "chemical bond" between Al_2O_3 and porcelain. This means that the external surface of the core does not need any retention elements. When using other veneer materials e.g. plastic, mechanical retention elements can be needed e.g. grooves, pits or on the external surface sintered retention elements or a silane treatment of the surface. The core can also be given such a shape that the ceramic inlay or onlay tooth crown does not need any veneer material. The surfaces of the inlay or the onlay tooth crown which is a part of the external surfaces of the repaired tooth must in this case before the cementing be ground and polished to a surface fineness of 0.5-5 μm preferably 0.5-1 μm .

The ceramic base material in the core comprises preferably one or several biocompatible oxides (including phosphates, silicates and sulfates), with the additives of carbides, silicides, nitrides or borides with or without binder metal (preferably iron-group metals). In addition to conventional sintering aids. The base material can also comprise other biocompatible high performance ceramics such as nitrides, oxynitrides, carbides, etc. Examples of the two former materials are Si_3N_4 , Si_2N_2O , sialon, AlN , $AlON$, etc. Examples of biocompatible oxides, which can form base matrix for the ceramic body, are Al_2O_3 , TiO_2 , MgO , ZrO_2 , and ZrO_2 (partly or totally stabilized with amounts of up to 25 weight% of Y_2O_3 , MgO or CaO).

Also, components such as SiC , TiN , TiC , TiB_2 , Si_3N_4 , or other biocompatible carbides or nitrides of group IV, V or VI can be present as particles with a size of <25 μm preferably <10 μm and/or as whiskers (hair shaped single crystals) with a length to diameter ratio >5, preferably >10 and/or fibers (polycrystalline) with a diameter

Pre -
sinter

Welding, i.
L... - y...
T... -